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भारतीय मानक

अण्विक चलनियाँ — विशिष्टि

Indian Standard

MOLECULAR SIEVES — SPECIFICATION

UDC 66.067.38

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

December 1994 **Price Group 3**

General Inorganic Chemicals Sectional Committee, CHD 03

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Molecular sieves are synthetically produced crystalline alkali metal alumino silicates of the empirical formula:

$$M_{2/n}O . Al_2O_3 . xSiO_2 . yH_2O$$

where M is the alkali metal cation with valency 'n' and the value of x and y vary with the alkali metal cation.

Molecular sieves are manufactured by crystallisation technique. Crystallisation of molecular sieves is achieved by close control of operating parameters, such as *p*H, temperature and pressure.

Molecular sieves are used in industry as desiccants/adsorbents, primarily intended for application in industrial air drying, gas and liquid drying, packing and in chromatographic analysis. This standard, however, does not cover the material intended for chromatographic use. For speciality applications, the additional requirements shall be as agreed to between the purchaser and the supplier.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MOLECULAR SIEVES — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for molecular sieves used as general purpose desiccants/adsorbents.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
460 : 1985	Test sieves (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
4905 : 1968	Methods for random sampling

3 TYPES

3.1 The material shall be of the following types:

a) Type 4A, 3A, 5A; and
b) Type 13 X.

4 REQUIREMENTS

4.1 Description

The material shall consist of crystalline alkali metal alumino silicate in granular, spherical or pellet form or as agreed to between the purchaser and the supplier.

4.2 X-ray Diffraction

The purity of the material shall also be evaluated by the X-ray diffraction pattern according to the method prescribed in A-2.

4.3 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 7 of the Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry and air-tight steel drums or as agreed to between the purchaser and the supplier. The containers shall not be opened until required for use and shall not remain open for a period more than required for taking out the material.

5.2 Marking

Each container shall bear legibly and indelibly the following information:

Table 1 Requirements for Molecular Sieves
(*Clause* 4.3)

Sl No.	Characteristics	Requirement Type				Method of Test Ref to Cl No. in Annex A
		4A	3A	5A	13X	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Moisture, percent by mass, *Max*	2.0	2.0	2.0	2.0	A-3
ii)	Adsorption capacity, percent by mass, *Min*	18.0	17.0	15.0	20.0	A-4
iii)	Thermal stability at 600°C (adsorption capacity, percent by mass), *Min*	18.0	17.0	15.0	20.0	A-5
iv)	Bulk density (of 1.5 mm and 3.0 mm pellets), g/ml, *Min*	0.78	0.78	0.78	0.68	A-6
v)	Loss on attrition, percent by mass, *Max*:					
	a) Soft surface (plastic)					
	1.5 mm pellets	0.15	0.15	0.15	0.15	A-7
	3.0 mm pellets	0.40	0.40	0.40	0.40	A-7
	b) Hard surface (steel)					
	1.5 mm pellets	0.80	0.80	0.80	0.60	A-8
	3.0 mm pellets	1.5	1.5	1.5	1.0	A-8
vi)	Bed crushing strength, percent by mass, *Min*	80	80	80	80	A-9

a) Name and type of the material;
b) Indication of the source of manufacture;
c) Mass of the material in the container;
d) Date of packing; and
e) Batch number.

5.2.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING AND CRITERIA FOR CONFORMITY

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(*Clause* 4.3 and *Table* 1)

METHODS OF TEST FOR MOLECULAR SIEVES

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 X-RAY DIFFRACTION

A-2.1 Instrument

A-2.1.1 *X-Ray Diffractomer*

A-2.2 Procedure

Grind the material and sieve through 63 micron IS Sieve (*see* IS 460 : 1985). Expose the sample to a constant atmosphere of 95 percent relative humidity. Pack it in a holder and scan on X-ray diffractometer at scanning speed of 1°/min. Typical X-ray diffraction intensities of the material have been shown in Table 2 obtained by using $Cu_{K\alpha}$ target, non-focal X-ray source of wavelength 1.541 8 (average of α_1 and α_2), and fixed divergent slit.

Table 2 X-Ray Diffraction of Molecular Sieves

Sl No.	Type 3A	Type 4A	Type 5A		Type 13x	
(1)	Intensity (2)	Intensity (3)	Intensity (4)	d(A°) (5)	Intensity (6)	d(A°) (7)
i)	*VVS*	*VVS*	*VVS*	12.29	*VVS*	14.465
ii)	*S*	*S*	*M*	8.71	*W*	8.845
iii)	*M*	*M*	*M*	7.11	*VW*	7.538
iv)	*W*	*W*	*W*	5.51	*MW*	5.731
v)	—	*VW*	—	4.36	*VW*	4.811
vi)	*M*	*MS*	*M*	4.11	*VW*	4.419
vii)	*S*	*S*	*MS*	3.714	*VVW*	4.226
viii)	*W*	*W*	*W*	3.417	*VVW*	3.946
ix)	*S*	*S*	*S*	3.293	*MS*	3.808
x)	*S*	*S*	*S*	2.987	*VVW*	3.765
xi)	—	*VW*	*VW*	2.904	*VVW*	3.500
xii)	*W*	*VW*	*VW*	2.754	*MW*	3.338
xii)	*M*	*M*	*M*	2.626	*VVW*	3.253
xiv)	*VW*	*VVW*	*VVW*	2.515	*VVW*	3.051
xv)					*VW*	2.944
xvi)					*MW*	2.885
xvii)					*VW*	2.794
xviii)					*VVW*	2.743
xix)					*VW*	2.664
xx)					*VVW*	2.620

Nomenclature:

VVS : Very very strong
VS : Very strong
S : Strong
MS : Medium strong
M : Medium
d : Interplaner spacing
MW : Medium weak
W : Weak
VW : Very weak
VVW : Very very weak
I : Intensity

NOTE — The peak intensities are measured by using divergent slit, whereas, in modern X-ray technique autodivergent slits are used with the result that the *d* values remain same but peak intensities will change.

A-3 MOISTURE

A-3.1 Apparatus

A-3.1.1 *Glass Weighing Bottle with Lid*

Flat bottomed of 20-ml capacity and preferably of the following dimensions:

Internal diameter	44 mm
Height	25 mm

A-3.2 Procedure

Weigh accurately 10 to 12 g of the sample directly from the packed container in a covered glass weighing bottle. Remove the lid and heat the bottle and its contents in an unventilated oven at 350 ± 5° C for 3 hours. Replace the lid, cool in a desiccator and weigh to constant mass.

A-3.3 Calculation

$$\text{Moisture, percent by mass} = \frac{100\,(M_1 - M_2)}{M_1}$$

where

M_1 = mass in g of the sample before heating, and

M_2 = mass in g of the sample after heating.

A-4 ADSORPTION CAPACITY

A-4.1 Apparatus

A-4.1.1 *Dial Hygrometer*

A-4.1.2 *Vacuum Desiccator*

A-4.1.3 *Vacuum Pump*

A-4.2 Reagents

A-4.2.1 *Saturated Solution of Lithium Chloride*

A-4.3 Procedure

Take about 5 g of the material in a previously weighed weighing bottle. Regenerate the sample by placing it in a furnace at 350°C for about 3 hours with a purge of dry air or nitrogen. Remove the sample and quickly stopper it and cool to 30°C in a desiccator without any desiccant. Weigh the sample bottle with the lid. Place a dish filled with saturated solution of lithium chloride and then the sample with the lid removed, in the desiccator, preferably a dial hygrometer. Evacuate the desiccator for about 15 minutes. Allow the sample to equilibrate for about 12 hours. Release vacuum and weigh the sample bottle with the lid.

NOTE — Saturated solution of lithium chloride at 30°C gives a relative humidity of 15 percent. Evacuation of the desiccator will increase the rate of adsorption and reduce the equilibrium time. It will be appropriate to place the desiccator at a constant room temperature.

A-4.4 Calculation

$$\text{Adsorption capacity, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of water adsorbed, and

M = mass in g of the regenerated material.

A-5 THERMAL STABILITY

A-5.1 Procedure

Heat 5 g of the material in a crucible at 600° C for one hour. Cover the crucible with a lid and cool it in a desiccator without any desiccant. Weigh the crucible with the lid and follow the procedure as stated in A-4.4 above, for determining water adsorption capacity, using saturated lithium chloride solution for adsorption.

A-6 BULK DENSITY

A-6.1 Procedure

Take the material exposed overnight to a constant atmosphere of 95 percent relative humidity and fill it in a 250-ml measuring cylinder up to 50 ml mark without knocking. Shake the sample down by gently lifting the cylinder as far as 5 cm from the base pad and releasing it on the table. Repeat this knocking for 4 more times, as above, after every 50 ml addition up to 250 ml. Level the cylinder with the material without any further knocking. Empty out the material from the cylinder and weigh to the nearest 0.5 g.

A-6.2 Calculation

Bulk density, g/ml = $0.004\,M$

where

M = mass in g of the material in the cylinder.

A-7 ATTRITION LOSS ON SOFT SURFACE (PLASTIC)

A-7.1 Apparatus

A-7.1.1 *Sieve* — 850 micron IS Sieve (*see* IS 460 : 1985).

A-7.2 Procedure

Weigh accurately 100 g of the material exposed overnight to a constant atmosphere of 95 percent relative humidity and tumble it in a plastic jar of 12 cm height and 10 cm diameter fitted with an edge of length 11.5 mm and thickness 1.5 mm, at 60 rpm for 45 minutes. Then measure the surface dust by sieving through 850 micron IS Sieve and express the dust collected as percent attrition loss.

A-8 ATTRITION LOSS ON HARD SURFACE (STEEL)

A-8.1 Apparatus

A-8.1.1 *Sieve* — 850 micron IS Sieve (*see* IS 460 : 1985).

A-8.2 Procedure

Weigh accurately 100 g of the material, exposed overnight to a constant atmosphere of 95 percent relative humidity and sieved through 850 micron IS Sieve. Fill the material 50 g each, into two steel cylinders of 30.5 cm length and 3.8 cm diameter. Clamp the cylinders at a distance of 7.6 cm from the axis of rotation and rotate at 30 rpm for 60 minutes. Sieve the material from both the cylinders through 850 micron IS Sieve and express the dust collected as percent attrition loss.

A-9 BED CRUSHING STRENGTH

A-9.0 General

Bed crushing strength is a measurement of the amount of fines formed on crushing under pressure.

A-9.1 Apparatus

A-9.1.1 *Cylinder*—of 150-ml capacity, inside diameter 45 mm closed at its bottom and fitted with a plunger.

A-9.1.2 *Press* — of 1.5 tonne.

A-9.1.3 *Sieve* — 850 micron IS Sieve (*see* IS 460 : 1985).

A-9.2 Procedure

Measure 120 ml of the material sifted out of the fines through 850 micron IS Sieve. Weigh the material. Place the material in the cylinder, insert the plunger and press slowly to 70 kg/cm^2. Pour carefully contents of the cylinder on to the same sieve and sift out the fines. Weigh the material retained on the sieve.

A-9.3 Calculation

$$\text{Bed crushing strength, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the material retained on the sieve, and

M = mass in g of the material taken for the test.

ANNEX B

(*Clause* 6)

SAMPLING AND CRITERIA FOR CONFORMITY OF MOLECULAR SIEVES

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampled material shall be kept in a protected place and shall not be exposed to damp air.

B-1.2 The samples shall be placed in suitable containers and each container shall be marked with full details of sampling giving the date of sampling; type of material; batch number, if any; and the name of the manufacturer.

B-2 SCALE OF SAMPLING

B-2.1 All the containers in a single consignment of the material of same grade drawn from a single batch of manufacture shall constitute a lot.

B-2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.3 The number of containers *n* to be chosen from the lot shall depend on the size of the lot *N* and shall be as given in Table 3.

Table 3 Scale of Sampling

(*Clause* B-2.3)

Sl No.	Lot Size *N*	No. of Containers to be Selected *n*
(1)	(2)	(3)
i)	Up to 15	3
ii)	16 to 50	5
iii)	51 to 100	8
iv)	101 and above	13

B-2.4 The containers to be selected for sampling shall be chosen at random from the lot. For this purpose a random number table shall be used (*see* IS 4905 : 1968). In case such a table is not available, the following procedure shall be adopted:

> 'Starting from any container, count them as 1, 2, 3 .... *r*, and so on in a systematic manner, where *r* is the integral part of *N*/*n*. Every *r*th container thus counted shall be withdrawn to constitute the required sample.'

B-3 PREPARATION OF SAMPLE

B-3.1 From each of the containers selected, draw approximately equal quantity of the material with the help of a suitable sampling implement. The material drawn from different containers shall be mixed thoroughly to give a composite sample which shall be sufficient to make triplicate determination for all the characteristics given in this specification.

B-3.2 The composite sample shall then be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

B-3.3 The referee sample bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two. It shall be used in case of a dispute between the two.

B-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-4.1 Tests for all the characteristics given in **4** shall be conducted on the composite sample.

B-4.2 The lot shall be considered as conforming to the requirements of this specification if all the test results on the composite sample satisfy the corresponding requirements given in **4**.

B-4.3 The requirement for X-ray diffraction shall be considered as a complimentary requirement

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